## Freeform Search

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DATE: Thu	irsday, February 19, 2004	Printable Copy	Create Case	
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	; PLUR=YES; OP=ADJ			0 r 11

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<u>L8</u>	L6 and nanofiltration	0	<u>L8</u>
<u>L7</u>	L6 and ananofiltration	0	<u>L7</u>
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<u>L1</u>	SPIRAL WOUND MEMBRANE AND DEAD END AND IMMERSED	4	<u>L1</u>

END OF SEARCH HISTORY

#### First Hit Fwd Refs

Generate Collection Print

L4: Entry 6 of 27

File: USPT

Apr 16, 2002

DOCUMENT-IDENTIFIER: US 6372143 B1

TITLE: Purification of produced water from coal seam natural gas wells using <u>ion</u> exchange and reverse osmosis

#### Abstract Text (1):

A method and apparatus are provided for treating produced water to lower the total dissolved solids of the water for safe discharge to the environment. The produced water is passed through a weak acid cation resin in H.sup.+ form to remove cations in the produced water and then preferably through a decarbonator to remove formed CO.sub.2 and to provide a first discharge stream. The cation loaded resin is then regenerated using a dilute acid, preferably diluted with produced water, to regenerate the resin to the H.sup.+ form. The resultant acid regenerated waste stream is discharged to a reject deep well. The acid regenerated resin is then rinsed, preferably with produced water, to remove residual salts to form a residual salt containing rinse stream. This salt stream is passed into a reverse osmosis unit to form a high salt containing reject stream which is discharged to a waste deep well and a low salt containing product discharge stream which may be safely discharged to the environment.

#### Brief Summary Text (5):

Pollution is a serious environmental problem for both industry and the public. Of particular concern are streams and lakes used as water supplies and government regulations limit the amount of various substances which may be discharged into and/or present in the water. In general, there are a number of different methods for the purification of water which include mechanical treatment by sedimentation, filtration or membranes; chemical purification by use of chemicals, gases or resins; and biological treatment by mixing water with bacteria to convert pollutants to innocuous byproducts.

#### Brief Summary Text (20):

rinsing the acid regenerated resin with water, preferably produced water, to remove residual <u>salts</u> from the acid regenerated resin forming a residual <u>salt</u> containing rinse stream;

#### Brief Summary Text (21):

passing the residual <u>salt</u> containing rinse stream into a separation unit to remove <u>salt</u> from the stream and preferably a <u>reverse osmosis</u> unit forming a <u>reverse</u> <u>osmosis</u> reject stream and a <u>reverse osmosis</u> product discharge water stream;

#### Brief Summary Text (22):

rejecting the  $\underline{\text{reverse osmosis}}$  reject stream (high  $\underline{\text{salt}}$ ) to a deep well or other environmentally safe reject site; and

#### Brief Summary Text (23):

discharging the  $\underline{reverse}$  osmosis product water discharge stream (low  $\underline{salt}$ ) to the environment.

#### Brief Summary Text (29):

rinsing the acid regenerated resin with water, preferably produced water, to remove residual salts from the acid regenerated resin forming a residual salt containing

#### rinse stream;

#### Brief Summary Text (30):

passing the residual <u>salt</u> containing rinse stream into a separation unit to remove <u>salt</u> from the stream and preferably a <u>reverse osmosis</u> unit forming a <u>reverse</u> osmosis reject stream and a <u>reverse osmosis</u> product discharge water stream;

#### Brief Summary Text (31):

rejecting the <u>reverse osmosis</u> reject stream (high <u>salt</u>) to a deep well or other environmentally safe reject site; and

#### Brief Summary Text (32):

discharging the  $\underline{\text{reverse osmosis}}$  product water discharge stream (low  $\underline{\text{salt}}$ ) to the environment.

#### Brief Summary Text (38):

means for adding rinse water, preferably produced water being treated, to the acid regenerated resin to form a residual salt containing rinse stream; and

#### Brief Summary Text (39):

a separation unit to remove <u>salt</u> from the stream and preferably a <u>reverse osmosis</u> unit to treat the residual <u>salt</u> containing rinse stream to form a <u>reverse osmosis</u> reject stream (high <u>salt</u>) for discharge to a deep well or other environmentally safe reject site and a <u>reverse osmosis</u> product water discharge stream (low <u>salt</u>) for discharge to the environment.

#### Brief Summary Text (43):

means for adding rinse water, preferably produced water being treated, to the acid regenerated resin to form a residual <u>salt</u> containing rinse stream; and

#### Brief Summary Text (44):

a separation unit to remove <u>salt</u> from the stream and preferably a <u>reverse osmosis</u> unit to treat the residual <u>salt</u> containing rinse stream to form a <u>reverse osmosis</u> reject stream (high <u>salt</u>) for discharge to a deep well or other environmentally safe reject site and a <u>reverse osmosis</u> product water discharge stream (low <u>salt</u>) for discharge to the environment.

#### Detailed Description Text (3):

Produced water contains high concentrations of various water-soluble <u>salts</u> (typically greater than 1000 ppm) usually typified by those present in sea water. Produced water may also commonly contain hydrocarbons and other dissolved water-soluble organic electrolytes.

#### Detailed Description Text (8):

A significant amount of residual <u>salt</u> is still present on the acid regenerated resin however, and a large amount of water is typically required to rinse ions such as sulfate ions (if H.sub.2 SO.sub.4 is used as the rinse water) from the resin in order to avoid contaminating new produced water with excessive amounts of sulfate after the <u>regeneration</u> process has been completed. The rinse water required to remove the sulfate from the regenerated <u>ion exchange</u> resin could be as much as .sup.30 % of the total volume of produced water treated.

#### Detailed Description Text (9):

To reduce this volume, it is an important feature of the invention that a two-step procedure be employed using first the dilute acid stream and then a produced water stream. The rinse water is segregated from the acid regenerated waste stream and the rinse water further concentrated with a reverse osmosis unit or other such salt separation unit. Accordingly, the rinse water, which is preferably produced water, is passed through the acid regenerated cation resin to form a residual salt containing rinse stream which stream is then passed through a salt separation unit,

e.g., a <u>reverse osmosis</u> unit. The reject stream from the <u>reverse osmosis</u> unit containing a large amount of <u>salt</u> is passed to a deep well as a <u>reverse osmosis</u> reject stream and the <u>reverse osmosis</u> product water discharge stream containing a low amount of <u>salt</u> may be passed safely to the environment.

#### Detailed Description Text (11):

Reverse osmosis membrane systems are used widely for purifying water such as desalinating seawater for potable water and irrigation purposes. For reverse osmosis membranes, a charge liquid containing a more permeable and a less permeable component is maintained and contacted under pressure with a non-porous separating layer. In a reversal of the cellular osmotic process, a portion of the charge liquid, predominately liquid, dissolves into the membrane and diffuses therethrough with a decreased concentration of salts. Usually, a substantial portion of the solute is left behind as the retenate and in the subject process as a reverse osmosis reject stream. Any suitable reverse osmosis membrane may be used in the method and apparatus of the invention and it is preferred to use a thin film composite because of its demonstrated effectiveness. Other suitable salt removal units may likewise be employed.

#### Detailed Description Text (15):

It is now necessary to rinse the acid regenerated weak acid cation resin to remove residual <u>salts</u> such as sulfate when using sulfuric acid as the regenerant. A rinse water 21, preferably produced water, is passed through the acid regenerated resin 11 to form a residual <u>salt</u> containing rinse stream 22. Stream 22 contains a large amount of sulfate ion due to the use of sulfuric acid as the acid resin regenerator. The residual <u>salt</u> containing rinse stream 22 is then passed through a <u>reverse osmosis</u> unit 23 to form a <u>reverse osmosis</u> reject stream 24 and a <u>reverse osmosis</u> product water discharge stream 25. The <u>reverse osmosis</u> reject stream 24 containing a high concentration of <u>salt</u> is then discharged to a deep well or other safe discharge 26. The <u>reverse osmosis</u> product water discharge stream 25 (low <u>salt</u>) may be safely discharged to a surface water stream 27.

#### Detailed Description Text (19):

From the above example it may be seen that produced water may be efficiently and cost effectively treated using the method of the invention. Thus, a produced water stream 10 having a TDS of about 6060 (#/day) is treated to form a first discharge stream 16 having a TDS of 180 (#/day) and a reverse osmosis procduct discharge water stream 25 having a TDS of 24 (#/day). Two waste discharge streams 19 and 24 are formed having a TDS of 5106 (#/day) and 3108 (#/day, respectively.

#### Detailed Description Paragraph Table (1):

TABLE 1 TDS TDS Stream No. Stream Name (ppm) #/day 10 Produced Water 1600 6060 12 Reduced dissolved solids 50 180 produced water 14 Air 177,000 15 CO.sub.2 3347 16 First Discharge Stream 50 180 18 5% H.sub.2 SO.sub.4 50 4087 19 Acid Regenerated Waste Stream 26,212 5106 21 Rinse Water 1600 1617 22 Residual Salt Containing Rinse 3100 3132 Stream 24 Reverse Osmosis Reject Stream 61,525 3108 25 Reverse Osmosis Product 25 24 Discharge Water

## <u>Current US Cross Reference Classification</u> (2): 210/651

#### CLAIMS:

1. A method for treating produced water to lower the total dissolved solids (TDS) of the water for safe environmental discharge comprising the steps of:

passing the produced water through a cation exchange resin in a H.sup.+ form to remove cations and form a first discharge stream and a solids loaded resin;

discharging the first discharge stream to the environment;

regenerating the solids loaded resin to a H.sup.+ form with a dilute acid solution to remove solids from the solids loaded resin into the dilute acid stream forming an acid regenerated waste stream and an acid regenerated resin;

rejecting the acid regenerated waste stream to a deep well,

rinsing the acid regenerated resin with water to remove residual <u>salts</u> from the acid regenerated resin forming a residual salt containing rinse stream;

passing the residual  $\underline{salt}$  containing rinse stream into a  $\underline{salt}$  separation unit forming a high  $\underline{salt}$  containing reject stream and a low  $\underline{salt}$  containing product discharge water stream;

rejecting the high salt containing reject stream to a deep well; and

discharging the low  $\underline{\mathit{salt}}$  containing product water discharge stream to the environment.

5. The method of claim 1 wherein the  $\underline{\text{salt}}$  separation unit is a  $\underline{\text{reverse osmosis}}$  unit.

# First Hit Fwd Refs End of Result Set

Generate Collection Print

L12: Entry 1 of 1

File: USPT

Mar 2, 1976

DOCUMENT-IDENTIFIER: US 3941693 A

TITLE: Solid mineral and regenerant recovery for ion-exchange resins

#### Abstract Text (1):

Recovery of ion-exchange regenerants and precipitation of mineral salts is disclosed. The spent cation and anion regenerant streams are combined to form a weak dissociable complex of the regenerants and to reconstitute the removed inorganic mineral salt. The mixture is adjusted stoichiometrically to balance the anion and cation of the removed salt. The salt is precipitated by limiting solubility thereof either by concentrating the mixture or by reducing the solubility of the salts therein, suitably by the addition of a water-miscible solvent such as methanol. The complex is dissociated to reconstitute the separate anion and cation regenerants which are adjusted to the appropriate solvent concentration for recycle and reuse. Excess water in the system provided by water of hydration released by the resin during regeneration or by washing steps is removed prior, during or after separation of the regenerants depending on the nature of the separation technique employed and the optimum time for precipitation and removal of the mineral salts. Separation and recovery of the regenerants may be accomplished by distillation of regenerants having disparate volatilities, solvent extraction using an immiscible solvent having preferential solubility for one of the regenerants or by forming an insoluble complex of one of the regenerants. The recovered regenerants are reused as regenerants for spent anion and cation resin columns and the precipitated slats are removed by filtration, sedimentation or centrifugation.

#### Brief Summary Text (5):

Water is becoming an increasingly scarce natural resource and with increasing population and industrial and commercial use of water, ionic pollution in the form of waste and by-product streams from industrial plants, refineries and municipal sewage is having a drastic effect on the ecological balance of lakes, rivers streams and even the oceans. Ionic pollution is found to destroy the food chain of marine life and in some cases so upsets the biological balance as to cause hazards to swimmers and fishermen. Even low ionic pollution in irrigation waters can have a drastic effect in terms of the accumulation of salts in the soil over extended periods of time. One of the main concerns of the present invention is to provide a method for demineralizing industrial and other waste streams prior to discharge into surface waterways. Water can be demineralized by many processes such as distillation, reverse osmosis, chemical precipitation or ion exchange. These processes, though all practiced commercially, suffer from limitations such as scaling, poor economics, and excessively high total dissolved solids (TDS) in the effluent, or the production of ecologically undesirable waste streams such as concentrated brines which are both expensive and difficult to dispose.

#### Brief Summary Text (6):

Since the development of commercially practicable synthetic <u>ion-exchange</u> resins, <u>ion-exchange</u> techniques have been the preferred methods for demineralizing water because of the high purity water produced, i.e., low TDS. However, the chemical costs for these systems have been quite high per unit of various salts removed. The

<u>ion-exchange</u> method which conventionally has been used consisted of the use of beds of strong acid cation exchanger in the hydrogen form. The resin must be regenerated with a strong acid or strong base, depending upon the nature of the chosen resin. In these prior art systems, regeneration requires a considerable excess of regenerant and the original reaction does not proceed to completion, even though the regeneration may.

#### Brief Summary Text (17):

A basic spent cation regenerant is combined with an acidic spent anion regenerant to form a weak dissociable complex of the regenerants and to reconstitute the mineral salt. The spent cation and anion regenerants are combined either in a continuous or batch fashion by blending near stoichiometric quantities of the materials. This stoichiometric adjustment is based on the amount of the two streams necessary to reconstitute the mineral salts to be removed from the <a href="ion-exchange">ion-exchange</a> resins. The regenerant streams may be either aqueous solutions or organic solvent water solutions. In either case, the regenerant streams are diluted by the amount of water of solvation released by the resin during regeneration and possibly by washes of the resin beds. This additional water is removed prior, during or after separation of the regenerants depending on the nature of the separation technique employed and the best time for precipitation and removal of the salts.

#### Detailed Description Text (6):

The separation technique to be employed depends on the specific <u>ion exchange</u> resins, the regenerants used to regenerate the resins and the particular salts which are to be precipitated.

#### Detailed Description Text (7):

The high degree of efficiency required of the process for it to be economical requires that not only the resin regenerations be essentially quantitative but that the separation and recovery of the regenerants be highly efficient. To achieve this separation it is necessary that the complex formed between the cation regenerant and the anion regenerant be sufficiently dissociated to provide enough of the anion regenerant for its rapid removal by distillation, complexation or solvent extraction. This separation becomes progressively more difficult as the acid concentration in the pot increases due to amine removal. Four requirements must thus be met by the regenerants: (1) the pK of the cation and anion regenerants must be low enough to assure quantitative regeneration of the respective spent ionexchange resins, (2) the pK of these regenerants must be high enough to provide adequate dissociation of their complex to permit ease of separation, (3) the regenerant heel remaining in the regenerant mixture after separation must not significantly interfere with the regeneration of the other resin bed or remain deposited on this resin bed after regeneration and (4) the regenerant to be separated must be stable to the separation media and must be stable under the conditions of the separation step. The pK of the regenerant will generally be between 3 and 6, preferably 5-7. Regeneration efficiency is related to the type of ion-exchange resin to be regenerated and the primary process of demineralization. The regenerant recovery process of the invention is generally adaptable to spent regenerant streams from separate or mixed beds of buffered or unbuffered weakly acidic cation exchange resins and weakly basic anion exchange resins such as those disclosed in U.S. Pat. No. 3,700,582 and in U.S. patent application Ser. Nos. 476,835 and 476,966, filed June 6, 1974. The term pK is utilized in the context as defined in the Condensed Chemical Dictionary, 8th Edition, Van Nostrand Reinhold Company, C 1971, at page 698.

#### Detailed Description Text (10):

The weak cation-exchange resins which are particularly useful in the practice of this invention are those which contain carboxylic acid groups. These resins can conveniently be obtained by the copolymerization of compounds such as acrylic acid, methacrylic acid, acrylic esters, methacrylic esters, acrylonitrile or methacrylonitrile, and other unsaturated acrylates or nitriles with appropriate

cross-linking agents such as divinylbenzene. If the resins are formed, however, from the polymerization of compounds containing ester or nitrile groups, hydrolysis to the corresponding acid must be effected prior to buffer addition. Further illustrations to the resins useful in the practice of the present invention and of methods for their preparation may be found by reference to U.S. Pat. Nos. 2,340,111; 2,371,818; 2,597,437; 2,885,371 and 2,963,453. In addition to the above-mentioned ionexchange resins, other weak-acid resins may be used. These resins may include those containing phosphonous, phosphonic, phosphinic or phosphoric acid groups and those resins which are made from phenol-formaldehyde condensates which also contain carboxylic acid groupings. The <u>ion-exchange</u> resins preferred at this time are those which contain either acrylic or methacrylic acid functional groups.

#### <u>Detailed Description Text (12):</u>

Weak "base" <u>ion-exchange</u> resins are generally resins having primary amine, secondary amine or tertiary amine as the principal functional group. Typically the weak base polyamines are copolymers of acrylonitrile and methyl acrylate crosslinked with divinylbenzene and then subjected to aminolysis with polyamines; copolymers of styrene-divinylbenzene chloromethylates treated with primary or secondary amines; and reaction products of phenolformaldehyde with a polyalkyleneamine. A weakly basic anion exchange resin may be defined as one which on titration with hydrochloric acid in water free from electrolytes has a pH below 7 when the amount of hydrochloric acid added is one-half the amount required to reach the inflection point (equivalence point).

#### Detailed Description Text (55):

As previously discussed, the choice of physical method of separating and <u>recovering</u> the <u>regenerants</u> influences the selection of regenerants. A more detailed description of particular separation techniques follows.

#### Detailed Description Text (120):

A pilot plant as shown in FIG. 9 capable of processing 1,000 gallons per day of waste water was operated as follows. The <u>ion-exchange</u> system consisted of a series of cation-anion resin bed sets identified as the carousel fixed beds buffered system as disclosed in application Ser. No. 476,835, entitled Buffered, Weak, <u>Ion-Exchange</u> Water Demineralization Process, filed June 6, 1974. The resins were CC-3 cation resins, IRA-68 anion resins, N-methylmorpholine as buffer at 40% of theoretical cation capacity, trimethylamine (2N) as anion regenerant and .beta.-methoxypropionic acid (2N) as cation regenerant.

#### Detailed Description Text (121):

The resin columns are all 6 inches inside diameter, the cation resin columns are loaded to a 27-inch bed depth (fully regenerated) with 0.442 cubic foot of CC-3 resin (54 g-eq theoretical capacity); the anion resins are loaded to a 54-inch bed depth (fully regenerated) with 0.884 cubic foot of IRA-68 resin (38.5 g-eq theoretical capacity). Eight columns were provided in five sets. The loading capacity of the lead anion resin column would be reached between 19 and about 29 hours with the system reducing 500 ppm in NaCl feed to water containing below 2 ppm NaCl with the column loaded to a level of 40%. Highest purity water is achieved when the ion beds have been essentially 100% regenerated. The anion resin is completely regenerated by using a 2N trimethylamine solution containing a 35% excess of regenerant; excess beyond a stoichiometric amount of anion loaded on the resin. The recycled cation regenerant composition is considerably more complex. The composition used successfully for regeneration is: 2.5 N MOPA acid, 0.8N TMA-MOPA acid salt, 0.4N sodium chloride, and 0.15N of unidentified ionized chlorides or chlorinated compounds. When the sodium chloride concentration approaches 0.8N and the TMA-MOPA nears 1.7N, the regeneration efficiency drops below 100%, sodium removal and cation bed performance suffers. The data demonstrates that with the use of TMA and MOPA as the regenerants, dissociation approaching 80% are regularly obtained at evaporator temperatures of 250.degree.F. At current ion-exchange loadings of 35% of theory, the regenerant recovery load is 0.1 gallon spent

regenerant for every gallon of water at 1,000 ppm NaCl processed. By increasing the <a href="ion-exchange">ion-exchange</a> loading to 70%, this load will be reduced almost inversely to 0.05 gallons regenerant per gallon process water at 1,000 ppm NaCl. This reduction results since the wash water represents the bulk of the spent regenerant volume and remains essentially constant regardless of ion bed loading. In addition, the cation regenerant volume remains constant since less excess regenerant is required as loading increases.

#### CLAIMS:

- 1. A method of recovering and recycling spent  $\underline{\text{ion-exchange}}$  regenerants comprising the steps of:
- 9. A method according to claim 1 further including the step of adjusting the concentration of the separated regenerants before recycling them to regenerate spent ion-exchange resin beds.
- 10. A method according to claim 1 in which the recycled regenerant is provided in at least a 30% stoichiometric excess with respect to ion loading of the ion-exchange resin bed.

### Hit List

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Search Results - Record(s) 1 through 5 of 5 returned.

1. Document ID: US 5460792 A

L11: Entry 1 of 5

File: USPT

Oct 24, 1995

US-PAT-NO: 5460792

DOCUMENT-IDENTIFIER: US 5460792 A

\*\* See image for Certificate of Correction \*\*

TITLE: Removal and destruction of halogenated organic and hydrocarbon compounds with porous carbonaceous materials

DATE-ISSUED: October 24, 1995

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Rosenbaum; Bruce M.

Ambler

PA

US-CL-CURRENT: 423/245.3; 210/763, 588/205

Full: Title:	Citation Fro	nt Review C	assification D	ate Refere	nce.	Claims	KOMC Drawii

2. Document ID: US 5308496 A

L11: Entry 2 of 5

File: USPT

May 3, 1994

US-PAT-NO: 5308496

DOCUMENT-IDENTIFIER: US 5308496 A

TITLE: Resin regeneration process

DATE-ISSUED: May 3, 1994

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Pease; Richard A.

Bear

DE

Rodini; David J.

Midlothian

VA

US-CL-CURRENT: <u>210/674</u>; <u>210/677</u>, <u>210/702</u>, <u>210/712</u>, <u>521/26</u>

Full Title Citation Front Review Classification Date Reference Claims KMC Draw D

h e b b g e e e f

3. Document ID: US 4207397 A

L11: Entry 3 of 5

File: USPT

Jun 10, 1980

US-PAT-NO: 4207397

DOCUMENT-IDENTIFIER: US 4207397 A

TITLE: Method for recovering and treating brine from water softener regeneration

DATE-ISSUED: June 10, 1980

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Davis; Stephen H.

Dayton

OH

Etzel; James E.

Lafayette

IN

US-CL-CURRENT: <u>521/26</u>; <u>210/676</u>, <u>210/677</u>, <u>252/184</u>

Full: Title: Citation Front Review Classification Date Reference Citation Claims KMC Draw De

4. Document ID: US 3941693 A

L11: Entry 4 of 5

File: USPT

Mar 2, 1976

US-PAT-NO: 3941693

DOCUMENT-IDENTIFIER: US 3941693 A

TITLE: Solid mineral and regenerant recovery for ion-exchange resins

DATE-ISSUED: March 2, 1976

INVENTOR-INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

DePree; David O.

Loomis

CA

US-CL-CURRENT: <u>210/674</u>

Full Title Citation Front Review Classification Date Reference

5. Document ID: US 3939071 A

L11: Entry 5 of 5

File: USPT

Feb 17, 1976

US-PAT-NO: 3939071

DOCUMENT-IDENTIFIER: US 3939071 A

TITLE: Recoverable regenerants for cationic exchange resins

DATE-ISSUED: February 17, 1976

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ef b

INVENTOR-INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Katzakian, Jr.; Arthur

Sacramento

CA

DePree; David O.

Loomis

CA

US-CL-CURRENT: 210/674; 210/685

Full: Title: Citation Front Review Classification Date Reference	Claims KWC Draw De
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recovering regenerant and ion exchange	5

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